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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART VII MAGNESIUM AND ITS ALLOYS AND THEIR EXAMINATION

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NEW DELHI 110002

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Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART VII MAGNESIUM AND ITS ALLOYS AND THEIR EXAMINATION

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Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART VII MAGNESIUM AND ITS ALLOYS AND THEIR EXAMINATION

0. FOREWORD

0.1 This Indian Standard (Part VII) was adopted by the Indian Standards Institution on 31 December 1975, after the draft finalized by the Metallography and Heat Treatment Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The primary object of metallographic examination is to reveal the constituents and the structure of metals and their alloys by means of a microscope. Because of diversity in available equipment, the wide variety of problems encountered and the personal element, this standard gives, for the guidance of the metallographer, only those practices which experience has shown are generally satisfactory.

0.3 This standard is being issued in parts. This part covers the polishing, etching and examination of magnesium and its alloys. The other parts of this code are as follows:

Part I	General features
Part II	Electrolytic polishing
Part III	Aluminium and its alloys and their examination
Part IV	Copper and its alloys and their examination
Part V	Iron and steel and their examination
Part VI	Lead and its alloys and their examination
Part VIII	Nickel and its alloys and their examination
Part IX	Gold, silver, platinum, palladium and their alloys
Part X	Tin and its alloys and their examination
Part XI	Zinc and its alloys and their examination

1. SCOPE

1.1 This standard (Part VII) covers the polishing, etching and examination of magnesium and its alloys.

2. PREPARATION OF SPECIMENS

2.0 Recommended methods of selection, size, cutting, cleaning and mounting of metallographic specimens are outlined in IS : 7739 (Part I)-1975*.

2.1 In general, the preparation of magnesium and magnesium alloy specimens is similar to the preparation of aluminium specimens. Sulphur or any of the plastic mounting materials may be used for mounting specimens too small to handle. Wood's metal or other metallic mounting materials should not be used because of galvanic attack at the point of contact when etched. The procedure described in **2.2** and **2.3** is recommended for developing the best results with magnesium but is not necessarily the only satisfactory method.

2.2 As in the preparation of aluminium specimens, a plane surface can be secured with a microtome; otherwise, grinding successively on aloxite cloths No. 50, 150 and 320 is recommended. The grinding is then finished by successive use of No. 0 and 000 metallographic emery paper. For convenience, the abrasive cloths or papers may be mounted on disks rotating at 800 to 1 200 rpm and the grinding on the No. 000 paper may be eliminated. (For details of grit numbers of abrasive papers, *see* IS : 715-1966† and IS : 2832-1964‡.) A comparative chart of grit numbers and trade designations is given in Appendix A.

2.3 Polishing is carried out in two stages. The rough polishing is done on ' Vel-chamee ' cloths mounted on disks rotating at 500 to 600 rpm. The abrasive used on this wheel is a distilled water suspension of No. 600 alundum. Only enough water is used to prevent seizure of the specimen. The fine polishing is accomplished on a velvet-covered or ' Vel-chamee ' covered disk rotating at 100 to 400 rpm and moistened with a suspension of specially levigated alumina (*see* Note). Seizure is prevented by using a small amount of filtered liquid soap on the wheel. After polishing, the specimen is rinsed in water, then in acetone or alcohol, and dried in a blast of warm air.

NOTE — The specially levigated alumina recommended for fine polishing can be easily prepared by placing 150 g of the best commercial levigated alumina and 2 litres of 0·001 N sodium hydroxide (NaOH) in a 4-litre bottle and agitating (by a stream of compressed air) for 30 minutes. Two more litres of 0·001 N sodium hydroxide (NaOH) are then added and the mixture shaken thoroughly. After standing for 1½ hours, the upper 12 mm of supernatant liquor is carefully siphoned off, yielding the solution for fine polishing. Additional 12 mm portions may be taken 1½ hours after each shaking.

*Code of practice for preparation of metallographic specimens: Part I General features.

†Specification for coated abrasives, glue bond (*second revision*).

‡Specification for waterproof silicon carbide paper.

3. ETCHING REAGENTS

3.1 In Table 1 are given the etching reagents commonly recommended for magnesium and its alloys.

3.1.1 The glycol etchant is suitable for almost all magnesium alloys in use today. Its virtue lies in its ability to reveal and outline the constituents without pitting or roughening. The technique of etching is as follows:

> Hold the specimen, polished face up, and immerse into the etchant with a sliding motion. Move the specimen back and forth during the time of immersion which will vary in time from 3 seconds on aged specimens to 1 minute on some solid solution alloys. Remove specimen and plunge in a stream of running water, then in acetone or alcohol and dry in a blast of warm air to avoid staining. The etched specimen should be preserved, if required, in a desiccator.

4. EXAMINATION AND IDENTIFICATION OF CONSTITUENTS

4.1 As is suggested in the description of etchants (Table 1), visual or low power macro-examination is usually made on specimens etched for ½ to 5 minutes in reagents No. 1, 2 and 3. For such examination the etching is deep and the polishing need not be as carefully done as is necessary for microscopical examination, where etching is usually done in dilute acids and for short times in order to secure the necessary detail and avoid coring. Although the etchants shown in Table 1 are the ones most commonly employed, numerous acids both organic and inorganic have been used with success. In all cases, etched specimens should be rinsed in running water, then in acetone or alcohol, and dried in a blast of warm air.

4.2 Table 2 outlines a method for identifying metallic constituents in sand-cast materials when viewed through a microscope using an Eastman No. 78A filter. In the wrought alloys the identification differs slightly, chiefly because of the increased solubility of certain constituents and because of spheroidizing and breaking up of constituents by heat treating or plastic deformation. The table indicates the condition (etched or unetched) under which the constituent is best identified, the minimum amount which can readily be detected, and the usual appearance. Many of the constituents appear in a globular form when present in small amounts but take a network form when larger amounts are involved. This change may occur with a difference of a few tenths percent for some of the elements, or it may require a change of as much as 10 percent for development.

TABLE 1 ETCHING REAGENTS FOR MAGNESIUM AND ITS ALLOYS

(*Clauses* 3.1 *and* 4.1)

Sl No.	Etching Reagent	Composition*		Remarks	Use
(1)	(2)	(3)		(4)	(5)
i)	Acetic acid	10 percent aqueous solution		Swab with cotton for ½ to 3 minutes	Macroetching
ii)	Tartaric acid	10 percent aqueous solution		Immerse polished face up	Macroetching flow lines in forgings
iii)	Acetic-picral	6 percent picric acid in 95 percent ethanol Glacial acetic acid	100 ml 10 ml	Immerse polished face up. Make etchant fresh just before using	Macroetching for grain size in solution heat-treated castings
iv)	Glycol etchant	Ethylene or diethylene glycol Distilled water Nitric acid (HNO_3)	75 parts 24 parts 1 part	*See* **3.1.1**	General etchant, excellent for alloys in the aged condition
v)	Acetic-glycol	Ethylene or diethylene glycol Distilled water Glacial acetic acid Nitric acid (HNO_3)	60 parts 19 parts 20 parts 1 part	Swab with cotton or immerse for 2 to 15 seconds	All wrought alloys and solution heat-treated cast alloys. Also good for Mg-Mn alloys
vi)	Phospho-picral	Ethyl alcohol (95 percent) Picric acid Orthophosphoric acid	100 ml 4 g 0·7 ml	Immerse face up until stained	For extreme contrast between compounds and solid solution

*The use of concentrated reagents is intended, unless otherwise specified.

TABLE 2 MICROSCOPICAL IDENTIFICATION OF CONSTITUENTS IN SAND-CAST MAGNESIUM ALLOYS

(*Clause* 4.2)

Element	Detection		Unetched		Etched with Glycol Etchant	
	Optimum Condition	Approximate Minimum Percent	Shape	Colour	Shape	Colour
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Al ($Mg_{17}Al_{12}$):						
Massive	Etched	2·0	—	—	Filigreed network‡	White
Precipitated	Etched	—	—	—	Lamellar or fine particles	Appears dark at low power
Zn ($MgZn_2$):						
Massive	Etched	1·5	—	—	Filigreed network‡	White
Precipitated	Etched	—	—	—	Fine particles	Appears dark at low power
Zn (Mg-AlZn):						
Massive	Etched	2·0	—	—	Massive‡	White
Precipitated	Etched	—	—	—	Lamellar or fine particles	Appears dark at low power
Mn	Unetched	—	†	Bluish-grey	Angular	Bluish-grey
	or					
	Etched	0·5	Angular	—	—	—
Cd*	—	—	—	—	—	—

(*Continued*)

TABLE 2 MICROSCOPICAL IDENTIFICATION OF CONSTITUENTS IN SAND-CAST MAGNESIUM ALLOYS — *Contd*

Element	Detection		Unetched		Etched with Glycol Etchant	
	Optimum Condition	Approximate Minimum Percent	Shape	Colour	Shape	Colour
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Si (Mg_2Si)	Unetched or etched	0·03	Plates or script	Light blue	Angular plates or script	Blue
Sn (Mg_2Sn)	Etched	4·0	Network	Blue	Filigreed or massive network§	Brown, dark blue, purple
Cu (Mg_2Cu)	Etched	0·5	—	—	Globular or network‡, ‖	White
Ni (Mg_2Ni)	Etched	0·5	—	—	Globular or network‡, ‖	White

*Cadmium is completely soluble in solid magnesium and its alloys. No constituent visible under microscope. Coring generally visible when etched.

†Manganese constituent very hard and polishes in relief. Usually appears as pits at low magnification and particles are resolved at a magnification of 250 × or more.

‡Outlined.

§Usually shows coring around constituent.

‖Depending on concentration.

APPENDIX A

(*Clause* 2.2)

COMPARATIVE CHART OF GRIT NUMBERS (APPROXIMATE) OF ABRASIVE GRAINS

Aluminium Oxide Silicon and Garnet		*Fliant*		*Class*		*Corundum*		*Emery*		*Trade Designation*
IS Grit Number	BS Grade Number	IS Grit Number	BS Grade Number	IS Grit Number	BS Grade Number	IS Grit Number	BS Grade Number	IS Grit Number	BS Grade Number	
14	14	—	—	—	—	—	—	—	—	—
16	16	—	—	—	—	—	—	—	—	—
24	24	24	3	24	3	24	—	24	—	Extra Coarse
30	30	30	2½	30	2½	30	—	30	—	Extra Coarse
36	36	36	2	36	—	36	—	36	3	Coarse
40	46	40	1½	40	S2	40	—	40	2½	Coarse
50	54	50	1	50	M2	50	—	50	2	Medium Coarse
60	60	60	½	60	—	60	—	60	1½	Medium
80	80	80	—	80	—	80	—	80	1	Medium
100	100	100	0	100	F2	100	—	100	F	Medium Fine
120	120	120	00	120	1½	120	—	120	FF	Fine
150	150	150	—	150	1	150	—	150	—	Fine
180	180	180	—	180	0	180	—	180	0	Extra Fine
220	220	—	—	—	—	—	—	—	—	—

NOTE — Grits 240 and finer come under the sub-sieve range and as limits for these cannot be set on common silk test sieves, the grain sizes shall conform to general commercial grading, and it is recommended that the sedimentation process be adopted for their analysis.

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Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95
Branch Offices::	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C- Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Patliputra Industrial Estate, PATNA 800013	26 23 05
Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005	32 36 35
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*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072	27 10 85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
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